National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2703

Sediment for Solid Sampling (Small Sample) Analytical Techniques

Standard Reference Material (SRM) 2703 is a marine sediment collected at the mouth of the Baltimore Harbor. SRM 2703 is primarily intended for use in evaluating analytical methods for the direct determination of selected elements in solid samples of marine or fresh water sediment and similar matrices. Direct and slurry sampling, as well as dissolution techniques using typically milligram size samples (<10 mg), can employ this SRM in the user's procedures; all certified and reference values are based on measurements using a samples size of at least 0.7 mg. Techniques using large samples (100 mg) should use SRM 2702 *Inorganics in Marine Sediment*. All of the constituents for which certified, reference, and information values are provided in SRM 2703 were naturally present in the sediment material before processing. A unit of SRM 2703 consists of a bottle containing 5 g of radiation-sterilized, freeze-dried sediment material.

Certified Concentration Values: A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values are based on the agreement of results from two or more chemically independent analytical techniques obtained at NIST and collaborating expert laboratories [1]. Certified values for concentrations, expressed as mass fractions, for 22 elements are provided in Table 1.

Reference Concentration Values: Reference values are non-certified values that are the best estimate of the true value. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1]. Reference values for concentrations, expressed as mass fractions, are provided for seven additional elements in Table 2.

Information Concentration Values: An information value is considered to be a value that will be of interest and use to the SRM user, but for which insufficient information is available to assess adequately the uncertainty associated with the value, or a value derived from a limited number of analyses [1]. Information values for concentrations, expressed as mass fractions, are provided in Table 3 for nine elements.

Expiration of Certification: The certification of this SRM lot is valid until **01 October 2014**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the investigations and technical measurements leading to the certification of this material was under the leadership of R. Zeisler of the NIST Analytical Chemistry Division.

Collection and preparation of SRM 2703 were performed by M.P. Cronise and C.N. Fales of the NIST Standard Reference Materials Program and B.J. Porter and M.M. Schantz of the NIST Analytical Chemistry Division. The sediment material was collected with the assistance of G.G. Lauenstein, J. Collier, and J. Lewis of the National Oceanic and Atmospheric Administration (NOAA).

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Consultation on the statistical design of the experimental work and evaluation of the data were provided by W.S. Ligget, Jr. of the NIST Statistical Engineering Division and D.L. Duewer of the NIST Analytical Chemistry Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

NOTICE AND WARNING TO USERS

Storage: SRM 2703 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Handling: This material is part of a naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicity. The particle size is in the range of respirable airborne particulate matter. Therefore, caution and care should be exercised during its handling and use.

INSTRUCTIONS FOR USE

Prior to removal of sub-samples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 2703 are reported on a dry-mass basis. The SRM, as received, contains approximately 2.3 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis, or a separate sub-sample of the sediment should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis. If the constituents of interest are volatile, then the moisture must be determined with a separate sub-sample. The drying procedures described below are recommended. Equivalent procedures may be used, but the temperature of 90 °C should **NOT** be exceeded.

PREPARATION AND ANALYSIS[1]

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from the Chesapeake Bay at the mouth of the Baltimore (MD) Harbor near the Francis Scott Key Bridge (39°12.3'N and 76°31.4'W). This location is very near the site where SRMs 1941 and 1941a *Organics in Marine Sediment* were collected. The sediment was collected using a Kynar-coated modified Van Veen-type grab sampler. A total of approximately 3300 kg of wet sediment was collected from the site. The sediment was freeze-dried, sieved at 70 µm (100 % passing), homogenized in a cone blender, radiation sterilized at 33 kGy to 45 kGy (^{60}Co) dose. A portion of this material has been issued as SRM 2702. The SRM 2703 portion (20 kg) was further processed by jet-milling and particle size classification to obtain a more narrow distribution of finer particles in the SRM 2703 for solid sampling analysis (Hosokawa Micron Powder Systems, Summit, NJ). The resulting material was homogenized again and then packaged in screw-capped amber glass bottles each containing approximately 5 g.

Particle Size Distribution: The particle size distribution for the parent stock material (SRM 2702) and the SRM 2703 was obtained using a commercial laser diffraction instrument manufactured by Malvern Instruments. The sediments were measured in aqueous suspension. The sediment suspensions were prepared by a 10 min sonication in 20 mL distilled water (with approximately 0.2 mg/mL of sediment with a drop of 0.1 % solution of Triton added). These suspensions were gradually introduced into the water-filled measurement cell until a 6.5 % obscuration of the laser beam was achieved. Each suspension was then measured three times for 30 s with 10 s pause between the passes. A refractive index of 1.52 and absorption index of 0.1 was selected for the measurements. Results were calculated using the General Purpose Model provided by Malvern (Malvern Master Sizer 2000). The results are shown in Figure 1.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 2703 are reported on a dry-mass basis; however, the material "as received" contains residual moisture. Drying in a conventional oven at 90 °C for at least 18 h is recommended. The measured moisture content was 2.27 % $\pm$ 0.09 % (95 % confidence level). Alternately, the amount of moisture in SRM 2703 can be determined by measuring the mass loss after freeze-drying. The following parameters for the freeze-drying method were successfully used for SRM 2702: 1.1 g to 1.3 g samples were lyophilized for four days at 1 Pa with a −10 °C shelf temperature and a −50 °C condenser temperature.

Homogeneity Assessment: The homogeneity of SRM 2703 was assessed by analyzing duplicate samples of approximately 0.8 mg from six bottles selected by stratified random sampling. Duplicate portions of each bottle and

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duplicate portions from six randomly selected locations in one bottle were analyzed by instrumental neutron activation analysis (INAA). Kurfürst homogeneity factors derived from these analytical data confirmed that a 1 % relative heterogeneity component of the uncertainty in the INAA results is not exceeded by selecting sample sizes of 0.7 mg to 1.5 mg. Laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS), micro-beam X-ray fluorescence (µXRF) and micro-beam proton induced X-ray emission (µPIXE) techniques complemented the INAA assay with homogeneity results for significantly smaller sample sizes.

Analytical Approach: In this investigation at NIST, INAA has been used as the solid sampling technique for the 0.7 mg size test portions and was supplemented with laser ablation - inductively coupled plasma mass spectrometry (LA-ICP-MS). Additional measurements with several solid sampling procedures and dissolution procedures were provided by collaborating laboratories with the following techniques: various modes of graphite furnace atomic absorption spectrometry (GFAAS), cold vapor atomic absorption spectrometry (CV-AAS), ICP-MS, X-ray fluorescence (XRF), and INAA. All assays were designed to establish commutability of values between the conventionally certified in SRM 2702, representing the parent material, and the SRM 2703 measured at small sample sizes. These measurements confirmed that the composition of the material had not changed in processing and that the measured values in SRM 2702 can be utilized for value assignment of SRM 2703.

Certified Values and Uncertainties: Certified values are derived from the analytical results used in the value assignment of SRM 2702 and the results of several analytical methods and laboratories on SRM 2703 for elements where agreement between the two materials was demonstrated in the small sample analysis. For consistency across elements, a Bayesian statistical model was chosen for computation of certified values and uncertainties [2]. The uncertainty listed with each value is an expanded uncertainty, with coverage factor 2 (approximately 95 % confidence). The results follow the ISO and NIST Guides [3].

For each element, there is a NIST result with an uncertainty that is complete in coverage of recognized sources of uncertainties, complemented by results from collaborating laboratories with similarly complete uncertainties, and usually several results without complete uncertainties. The uncertainties of the latter results were augmented on the basis of the differences among the results obtained by different methods [4]. The Bayesian methods combine results by different methods from different laboratories based on the results provided and the uncertainties provided with them. Thus, a consistent methodology was used for all elements.

Table 1. Certified Concentrations for Selected Elements

Elements	Mass Fraction (mg/kg, unless noted as %)			Elements	Mass Fraction (mg/kg, unless noted as %)		
Al [b,c,f,G,g]	8.33 %	±	0.22 %	Na [b,c,f,G,g]	0.693 %	±	0.019 %
As [b,c,d,G,g]	45.5	±	1.7	Pb [A,a,b,c,f,L]	130	±	11
Ba [A,b,d,f,g]	416	±	32	Rb [b,f,G,g]	130	±	11
Cd [A,a,b,d,I]	0.811	±	0.076	Sb [a,b,f,G,g]	5.62	±	0.26
Ce [b,c,f,G,g]	125.5	±	5.0	Sc [b,c,G,g]	25.95	±	0.68
Co [b,c,d,G,g]	27.70	±	0.50	Sr [A,b,c,f]	118	±	18
Fe [b,c,f,G,g,k]	7.38 %	±	0.32 %	Th [b,c,f,G,g]	20.22	±	0.74
Hg [A,e,H]	0.474	±	0.066	Ti [b,c,f,G,g,k]	0.880 %	±	0.046 %
K [b,c,f,G,g]	2.08 %	±	0.24 %	U [b,f,G,g]	8.99	±	0.72
La [b,c,f,G,g]	75.9	±	3.0	V [b,c,G,g]	360	±	13
Mn [b,c,G,g,k]	1734	±	48	Zn [a,b,c,f,G,g]	480	±	22

Analytical techniques used for certified values (Capital letters indicate that method was used by NIST):

A, a   Inductively Coupled Plasma Isotope Dilution Mass Spectrometry (ICP-ID-MS)
b   Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
c   Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
d   Graphite Furnace Atomic Absorption (GFAAS) and Flame Atomic Absorption Spectrometry (FAAS)
e   Cold Vapor Atomic Absorption Spectrometry (CV-AAS)
f   X-ray Fluorescence (XRF)
G, g   Instrumental Neutron Activation Analysis (INAA)
H   Radiochemical Neutron Activation Analysis (RNAA)
I   Prompt Gamma Activation Analysis (PGAA)
k   Micro X-ray Fluorescence (µ-XRF)
L   Laser Ablation ICP-MS

NOTE: INAA and GFAAS procedures established equivalence of SRM 2703 with SRM 2702, therefore the data from both materials were used for value assignment.

Reference Values and Uncertainties: Reference values are based on results from one method carried out in several laboratories or from two or more analytical methods without NIST results. The Bayesian methods of combining the results by different methods from different laboratories were applied as above. These results do not fulfill the criteria for certification since a full estimate of method bias or results from NIST methods have not been available. The reporting follows the ISO and NIST Guides [3].

Table 2. Reference Values for Concentrations of Selected Elements

Elements	Mass Fraction (mg/kg, unless noted as %)			Elements	Mass Fraction (in mg/kg, unless noted as %)		
Ag [a,d]	0.59	±	0.12	Hf [G,g]	11.8	±	0.8
Ca [b,c,f,g]	0.31 %	±	0.12 %	Sm [G,g]	10.8	±	0.8
Cs [b,G,g]	7.7	±	0.7	W [G,g]	6.4	±	0.6
Cu [a,b,c,d,f]	120	±	15				

Analytical techniques used for reference values (Capital letters indicate that method was used by NIST):

a   Inductively Coupled Plasma Isotope Dilution Mass Spectrometry (ICP-ID-MS)
b   Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
c   Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
d   Graphite Furnace Atomic Absorption (GFAAS) and Flame Atomic Absorption Spectrometry (FAAS)
f   X-ray Fluorescence (XRF)
G, g   Instrumental Neutron Activation Analysis (INAA)

NOTE: INAA and GFAAS procedures established equivalence of SRM 2703 with SRM 2702, therefore the data from both materials were used for value assignment.

Information Values: Information values are given in SRM 2703 to assist users in the assays of non-certified elements. Information values are based on results that did not allow complete assessment of all sources of uncertainty; therefore, only estimated means without uncertainties are given.

Table 3. Information Values for Selected Elements

Element	Mass Fraction (in mg/kg, unless noted as %)	Element	Mass Fraction (in mg/kg, unless noted as %)
Mg [b,c,f,g]	1.0 %	P [b,c,f,H] (%)	0.16 %*
Mo [b,c,f]	11*	Se [b,c,f]	4.9*
Nb [c,f]	63*	Sn [a,c,f]	32*
Nd [c,f,g]	72	Tl [A,a]	0.83*
Ni [A,a,b,c,d,f]	75*		

*The element was not determined in SRM 2703. The information value is given based on the determinations carried out on the parent material SRM 2702.

Analytical techniques used for information values (Capital letters indicate that method was used by NIST):

A, a   Inductively Coupled Plasma Isotope Dilution Mass Spectrometry (ICP-ID-MS)
b   Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
c   Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
d   Graphite Furnace Atomic Absorption Spectrometry (GFAAS)
f   X-ray fluorescence (XRF)
H   Radiochemical Neutron Activation Analysis (RNAA)

SUPPLEMENTAL INFORMATION

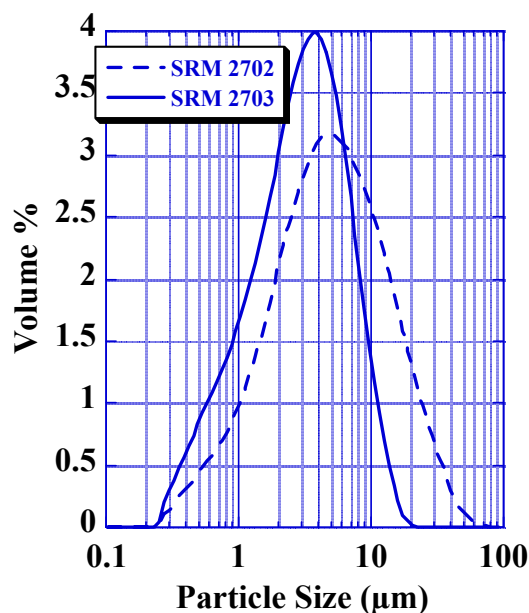**Particle Size Distribution**

Figure 1. Particle size distributions in SRM 2702 (parent material) and SRM 2703 determined in aqueous suspension via laser light scattering instrumentation (Malvern Mastersizer 2000). Calculated 10, 50, and 90 percentile particle sizes (percent volume of particles smaller than the value) for SRM 2703 are: $d_{0.1} = 0.89$ µm, $d_{0.5} = 2.87$ µm, $d_{0.9} = 7.25$ µm (SRM 2702: $d_{0.1} = 1.19$ µm, $d_{0.5} = 4.11$ µm, $d_{0.9} = 14.3$ µm). Uncertainties in these values are estimated at ± 10 % relative (2s).

Homogeneity Assessment

Micro-PIXE Imaging Analysis: Samples were analyzed using a 3 MeV focused proton beam. From each of the five bottles, one pellet was prepared for µ-PIXE analysis. Micro-PIXE images were collected for all elements found in the PIXE spectrum. Only sulfur showed heterogeneity directly observable from the 2D maps in all SRM 2703 samples. Elements with enough counting statistics were considered later for the analysis. Samples were scanned first over 750 µm × 750 µm area. This scan area was divided in of-line analysis to 8 regions of 187.5 µm × 375 µm each (Figure 2). Spectra from each of 8 smaller regions were separately analyzed (sample 1 through 8). X-ray line intensities were found using the program GUPIX and the same input parameters were used to fit all spectra.
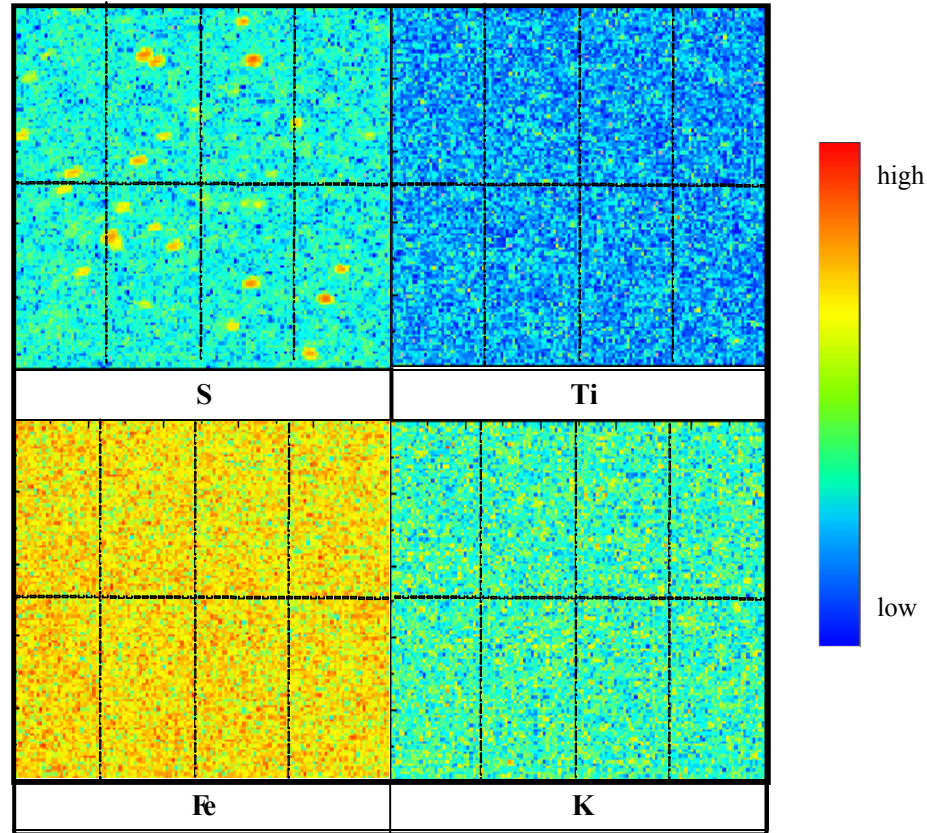

Figure 2. Micro PIXE images of the SRM 2703 (bottle #1291) sample. Total scan size of 750 µm × 750 µm is divided in eight equal regions.

The quantitative evaluation of the scanned areas is shown for the scans of the SRM 2703 bottle #1291 sample in Table 4. All five sample scans produced similar results. All results are given as the relative standard deviations u_{HET} (contribution from heterogeneity) that is derived from the measured total standard deviation u_{exp} and its analytical contribution u_{AN} according to:

$$u_{\text{exp}}^2 = u_{\text{HET}}^2 + u_{\text{AN}}^2 \tag{1}$$

Since the X-ray intensities are attenuated in the sample, the highest contribution comes from the surface sample layers. The analyzed sample mass varies for each element and was calculated from a sample thickness that contributes to 90 % of the X-ray yield. To obtain sample mass, the calculated thickness was multiplied with the sample area exposed to the ion beam and multiplied with the sample density. The data show acceptable homogeneity for the measured elements at the smaller than 10 µg sample size, with less satisfactory results for sulfur and calcium.

Table 4. Results of the Nuclear Microprobe PIXE Scan Over the
Pellet of Sample SRM 2703 (bottle #1291)

Sample	Al	Si	S	Cl	K	Ca	Ti	Mn	Fe
1	65752	219170	12146	2419	15985	2643	6958	1052	44221
2	64129	218360	13589	2465	15892	2524	6828	1059	43295
3	62936	217710	11989	2253	15421	2353	6622	988	42940
4	61812	216010	12961	2314	15246	2416	6533	1112	42538
5	63205	218770	12088	2291	15671	2634	6665	1109	43290
6	62759	217320	13591	2408	15523	2552	6696	1142	42862
7	62376	216260	12825	2247	15484	2559	6457	1032	41911
8	61114	214380	10254	2355	15134	2336	6324	1031	42180
I_{ave} (counts)	63010	217248	12430	2344	15545	2502	6635	1066	42905
u_{exp} (%)	2.3	0.7	8.7	3.5	1.9	4.8	3	4.8	1.7
mass (μg)	1.0	1.6	1.6	2.3	3.9	4.8	6.1	7.0	7.2
u_{HET} (%)	2.3	0.7	**8.6**	2.8	1.7	**4.4**	2.7	3.6	1.6

NOTE: The total scan was divided to 8 regions each 187.5 μm × 375 μm. X-ray intensities for elements with significant statistics are given in counts for the each region.

INAA Determinations: INAA has been shown to be applicable for the determination of heterogeneity in small samples because the small samples, which essentially form point sources, provide for great improvements in the assays [5]. Based on the experimental model that the observed experimental variance of the INAA results (u^2_{exp}) was the summation of variances due to heterogeneity (u^2_{HET}) and the relevant analytical components (u^2_{AN}), the variances from heterogeneity can be calculated (Eq. 1). In the case of this INAA procedure the analytical variance is in many instances dominated by the uncertainty from counting statistics.

Based on the models linking sample mass (w) to the homogeneity of particulate materials [6], Kurfürst et al. have proposed an elemental homogeneity factor H_e that gives the relative standard deviation in percent for the element of interest if 1 mg samples were repeatedly analyzed and no analytical uncertainty was to influence the result (Eq. 2) [7].

$$H_e = u_{HET} \sqrt{w} \tag{2}$$

Twelve test portions of SRM 2703 were analyzed and the heterogeneity components were calculated by subtracting the analytical uncertainties from the observed experimental uncertainty. The results for elements with counting statistics of 1 % relative or smaller are shown in Table 5. The Kurfürst homogeneity factors are very similar for these elements and reveal homogeneity for the observed elements at sample sizes of 1 mg or smaller.

Table 5. Homogeneity Test Results Obtained by INAA for Small Samples of SRM 2703

Element	Mass (mg)	Observed Uncertainty	Counting Uncertainty	Other Uncertainties (estimate)	Uncertainty Due to Heterogeneity	Kurfürst Homogeneity Factor ($\sqrt{m}$)
Al	0.8	0.92	0.37	0.5	0.68	0.61
As	0.7	2.22	0.84	0.7	1.93	1.62
Ce	0.7	1.17	0.91	0.5	0.54	0.45
Co @ 1723 keV	0.7	1.26	0.89	0.5	0.74	0.62
Co @ 1332 keV	0.7	1.33	0.88	0.5	0.86	0.72
Fe @ 1099 keV	0.7	1.26	0.77	0.5	0.86	0.72
Fe @ 1292 keV	0.7	1.09	0.72	0.5	0.65	0.54
La @ 487 keV	0.7	1.30	0.79	0.7	0.76	0.63
La @ 1596 keV	0.7	1.39	0.78	0.7	0.91	0.76
Mn @ 1811 keV	0.8	1.08	0.50	0.7	0.65	0.58
Mn @ 2113 keV	0.8	1.04	0.66	0.7	0.39	0.35
Na @ 1368 keV	0.8	1.21	0.87	0.7	0.47	0.42
Na @ 2754 keV	0.8	0.80	0.79	0.7	-*	-*
Sc @ 889 keV	0.7	1.00	0.99	0.5	-*	-*
Sc @ 1121 keV	0.7	1.16	0.99	0.5	0.34	0.28
Th	0.7	1.51	1.03	0.5	0.98	0.82
V	0.8	1.49	0.90	0.5	1.08	0.96
Zn	0.7	1.31	1.10	0.5	0.51	0.42

NOTE: Reported are elements with low counting uncertainties. Uncertainties are % relative values (1 standard deviation) for each component.

*Factor could not be calculated because the observed experimental uncertainty is similar to the analytical uncertainty.

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Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.